SISAL FIBRE AND ITS APPLICATIONS

SUBMITTED BY

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UNDER THE GUIDANCE OF

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Supervisor's Certification

This is certify that the work in the thesis entitled ***"Sisal fibre and its applications"*** submitted by ***Abhishek Ray*** is a record of original research work and has been carried out under my supervision and guidance in partial fulfilment of the requirements for the award of the degree of ***Master of Science in Physics*** at National Institute of Technology, Rourkela. The work is satisfactory and has not been submitted elsewhere before for any other academic degree or award to the best of my knowledge.

Place: Rourkela Prof. D.K Bisoyi

Date: 1.7.16

Dedication

I dedicate this thesis to my dreams and ideology.

~Abhishek Ray

Declaration

I, Abhishek Ray, Roll Number: 414ph2050 hereby declare that the thesis entitled ***"Sisal fibre and its applications"*** submitted to NIT, Rourkela is a record of my original research work under the supervision of Prof. Dillip Kumar Bisoyi, Associate Professor, Department of Physics and Astronomy, NIT, Rourkela, Odisha.

I am fully aware that in case of any non-compliance detected in future, the Senate of NIT Rourkela may withdraw the degree awarded to me on the basis of the present dissertation.

Place: Rourkela

Date : 1.07.16 Abhishek Ray

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Last but not the least I am extremely thankful to my parents who were always there to help and support me.

ABSTRACT

Sisal fibre is treated with alkali for the improvement of the fibre properties after being dewaxed and followed by vacuum oven drying. In order to measure all the effects on the sisal fibre composite due to the alkali(here KOH) treatment , various characterizing analyses such as XRD,SEM, FTIR and 3 point flexural tests are carried out. As a result of the treatment, the improvement on the surface of the fibre is observed due to the reduction of the impurities, lignin content etc , which is confirmed by FESEM analysis an increase in fibre strength is also obtained which may be due to better fibre-matrix linkage. A decrease in degree of crystallinity is observed during XRD analysis. However an increased physical and mechanical properties is observed due to the alkali treatment on the sisal fibre. Optimally 3% treated sample comes out to be the sample of choice.

Keywords : sisal, alkali, matrix, FESEM, XRD, FTIR, Flexural test, composite

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CHAPTER 1

INTRODUCTION

Sisal fibre and its brothers-in-arms e.g. bamboo, jute, coir etc. came into the broader picture when alternative sources to timber were being searched. There has been many peekings into the alternate approach and polyethylene compounds e.g plastic were considered the forerunner for filling up the void. Pipes, heavy industry, building industry all rely on plastics upto a great deal.

However it has also given rise to another threat- shortage of plastics in the future, leading to the idea of plastic filled composites. And as a result , reinforcement of the plastic material using natural fibres started getting notice. Different parts of plants and fruits of many crops have been found to be viable sources of raw material for industrial purposes due to high performance in some mechanical based properties, dielectric properties, post-processing advantages and low cost. These materials have been earmarked as a potent recipe of the future due to the good potential lying as a substitute of wood in structural applications.

Fig.1 Short sisal fibre

Coir is a tough multicellular fibre with a central portion called “lacuna”.

Sisal is a very strong leaf fibre.

Elementary unit of a cellulose macromolecule is anhydro-d-glucose, which contains 3 alcohol hydroxyls(-OH) (Bledzki et. Al 1996)

All plant fibres are of hydrophilic nature; their moisture content reaches 8-13 %

Sisal became an important part of the fibre revolution after prowessing high impact strength besides having moderate tensile and flexural properties compared to lignocelluloses.

Use of sisal as a textile fibre by mankind began with Weildling in 1947. Along with the study of agronomic and industrial aspects, a through and fundamental investigation on sisal fibre was carried out by Wilson in 1971. He also pointed out the possibilities of chemically modifying the fibre.

From then on, several studies have reported on the use of sisal fibers as reinforcements in polymer matrices. Considerable work was done by Barkakaty in 1976, Bisanda and Ansell in 1991, Joseph et al in 1992, Mattoso et al in 1997.

STRUCTURE AND PROPERTIES OF SISAL FIBRE

Sisal is a fibre obtained from the leaves of the plant Agave Sisalana. A native of Mexico, it is now maintained and cultivated in East Africa, Brazil, Haiti , India mainly in Assam and Indonesia.

Weindling grouped sisal under “hard fibres” coming only behind manila in durability and strength.

The name ‘sisal’ comes from a harbour town in Yucatan in Maya, Mexico meaning cold water.

A healthy sisal plant produces nearly 200-250 leaves roughly composed of 1200 fibre bundles per leaf containing nearly 4 % fibre,0.75 % cuticle, 8% dry matter and 87.25 % water. So, a normal leaf will yield around 3% w/w fibre.

Sisal leaf consists of 3 types of fibres:-

1. Mechanical
2. Ribbon
3. Xylem

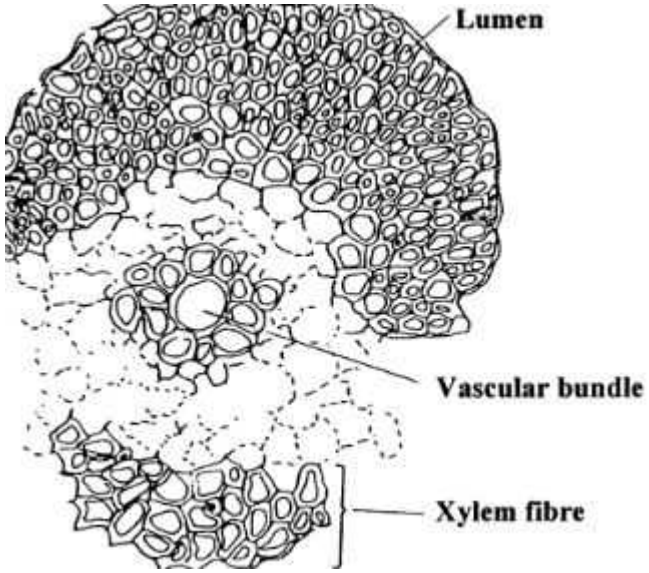


Figure 2 showing detailed cross section

Mechanical fibres are mostly extracted from periphery of leaf. They have a roughly thickened-horseshoe shape and seldom divide during extraction processes. They are the most commercially useful of the sisal fibre.

Ribbon fibres occur in association with the conducting tissues in the median of the leaf. The related conducting tissue structure of the ribbon fibre gives them considerable mechanical strength. They are long and compared with mechanical fibres they can be split easily in longitudinal direction during processing.

Xylem fibres comes in indefinite shapes and are located opposite to ribbon fibres through the connection of vascular bundles. Mainly comprising of thin-walled cells, they are easily breakable and sometimes lost during the extraction process.

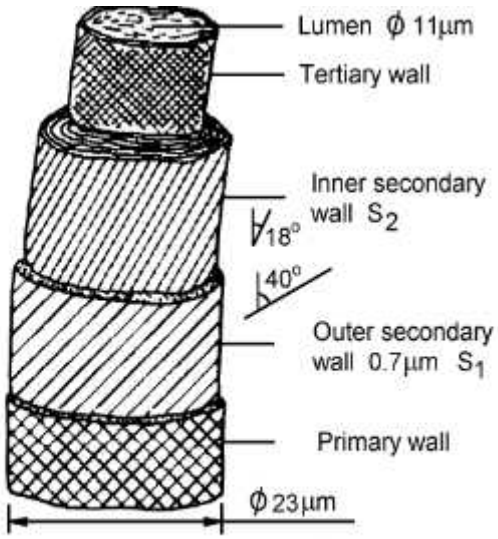


Figure 3. Schematic sketch of a sisal fiber cell with approximate dimensions (Gram, 1983)

Major studies on sisal fibres carried out during the past 2 decades mainly focussed on the following topics :-

1. **Properties of sisal fibres**

Mechanical, thermal and dielectric properties of sisal fibre have been studied using X-ray diffraction, Infrared, TG, SEM, DSC, DMA etc. have been used to determine the characteristics of sisal fibre and provide theoretical support for processing and application of this fibre.

Properties of plant fibres depend on the cellulose content and the spiral angle which bands of microfibrils in the inner secondary cell wall make with the fibre axis.

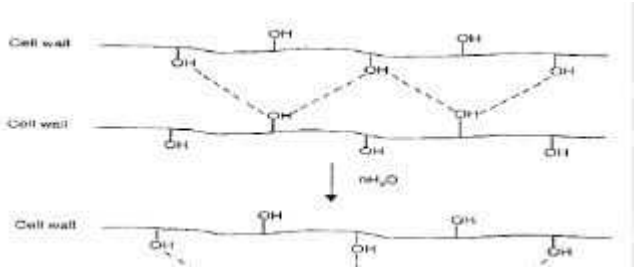


Figure 4 showing illustration of the swelling process in cellulose

Tensile strength, modulus and toughness (defined as energy absorption per unit volume) of sisal decrease with increase in temperature (Chand and Hashmi).

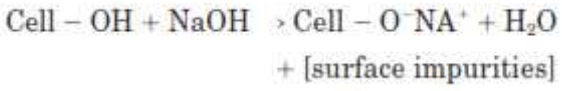
For electrical applications, the dielectric properties of sisal fibre at different temperatures and frequency vary reciprocally. With an increase in frequency, the dielectric value decreases proportionally, whilst for temperature it has an increasing nature. As Yan Li reports in 2000, plant age shifts dissipation factor (tan δ) peak to higher temperatures. He used IR, XRD and TG for studying the thermal properties. IR spectrum didn't change below 200°C treatment but density and crystallinity increased. They also concluded there are 3 steps related to thermal degradation.

2. Interface properties between fibre and matrix

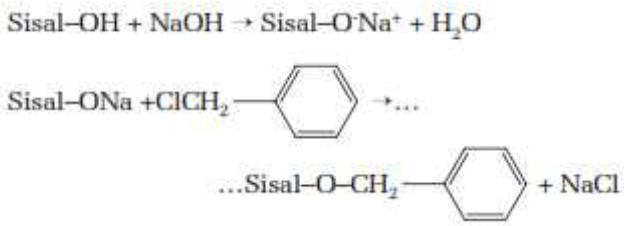
By using chemical and thermal treatment methods, bond strength between fibre and matrix can be increased and reduction of sisal fibre. Interface mainly determines hydrophilicity but some polymer matrices maybe hydrophobic leading to poor bonding with sisal.

Yang et al studied the relationship of surface modification and tensile properties of sisal. The major modification methods include :-

1. Alkali treatment



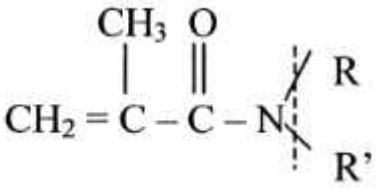
2. H_2SO_4 treatment
3. Conjoint H_2SO_4 and alkali treatment
4. Benzol/Alcohol dewax treatment



5. Acetylated treatment
6. Thermal treatment
7. Alkali-Thermal treatment
8. Thermal-Alkali treatment

Thermal treatment(at 150^{o}C for 4 hrs) was found out to be the most desirable method for increasing strength and modulus properties because of increased crystallinity.

Use of coupling agents like N-substituted methacrylamide



results in great reduction of moisture absorption of surface treated fibres by inviting hydrophobicity to the surface via long-chain hydrocarbon attachment

3. **Properties of sisal-fibre reinforced composites**

Majorly fibre length, fibre orientation, physical properties etc. are studied after sisal has been reinforced with its matrix, which maybe organic or inorganic resulting in samples having improvised mechanical properties (as in case of thermoplastics) or a boost to fire and environment change resistances (as in case of gypsum)

Majorly the matrices widely used are :-

a. Sisal-fibre reinforced thermosets(e.g Polyster)
b. Sisal-fibre reinforced thermoplastics(e.g Polyethylene, Polystyrene, PVC)(2 distinct ways namely (a) Melt mixing (b) Solution mixing)
c. Sisal-fibre reinforced rubber matrix (e.g Natural rubber, Styrene-butadiene rubber)

d. Sisal-fibre reinforced cement and gypsum matrix accounts for the worst interface between sisal and matrix composites as the fibres absorb moisture from cement. Swift went to mark this out as a potential player for rural Africa.
e. Sisal/glass-fibre reinforced hybrid composites utilise the properties of both and are added conjointly for the production of an optimal, superior but economically viable composite.
f. Other matrix systems(e.g Sisal-reinforced CSNL-Cashew Nut Shell Liquid)

4. EXTRACTION OF SISAL FIBRE

Sisal fibres are extracted from its leaves very carefully using methods as suggested by Chand and Mukherjee & Satyanarayana, include :-

a. Retting followed by scrapping
b. Mechanical means using decorticators

Amongst these two, mechanical process is more superior as it yields about 2-4 % fibre (approx. 15 kg per 8 hr) with good quality and lustrous colour, while the one produced by the former process yields poor quality fibres with dull colour. After extraction, fibres are washed in clean water and/or distilled water to remove surplus wastes like chlorophyll, leaf juices and adhesive solids.

CHAPTER 2

EXPERIMENTAL WORK

The Sisal used in the lab work was obtained from the Sisal Research Station, Indian Council of Agriculture Research, Bamara , Odisha having a diameter of 170-300 micrometer. The density of the fibre was nearly 1.5 g/cm^3.

1. Treatment Processes

a. Initially fibers were cut into smaller pieces and washed with surf water (2 % w/v), scrubbed and was treated likewise for 24 hours. This process aims at removing the dirt and dust particles from the outer surface of the fiber.
b. Next the surf-treated sisal fibers were taken in batches of 25g , soaked in 400 ml Benzene and 200ml alcohol(2:1) mixture(600 ml mixture)to participate in a process called dewaxing which is discussed later.

As sisal fibers are derived from lignocelluloses, which contain strongly polarised –OH group. The surface of the fibre is coated with waxy substances and having lower surface tension. Bond strength can be improved by

a. Dissolving fatty substances and layer of cuticle from fibers.
b. Making the fibre hydrophobic by reacting with some reagent.
c. Increasing the compatibility of the fibre with the resin by grafting it with some suitable polymers.

Dewaxing involves immersing the fibers in the ethanol and benzene mixture solution for 12 hours with 2 hours of intermediate heating and alternate cooling of solution in which fibers were soaked. The heating was started from 30 degree centigrades and heated upto 55 degree centigrades and then maintained for 2 hour span. Correspondingly its lowered to 30 degree centigrades and maintained for 2 hours. The dewaxed fibre removes surface impurities like

natural wax, lignin, hemicelluloses and removal of internal constraint from the fibre. Dewaxing of fibre helps in improving fibre matrix. Its also possible to increase the moisture content of sisal fibre

The next step following this chemical dewaxing process is Alkalisation. **Alkalisation** refers to treatment of fibers using alkalis like NaOH, KOH, LiOH resulting in reportedly better surface modification and leading to better mechanical properties like tensile strength, breakage strength, thermal properties, crystalline properties. Generally crystallinity increase for sisal, jute and kapok fibres. Also fiber-resin adhesion increases, resulting in increased inter-facial energy and improvement in mechanical and thermal stability of the composites.

Most of the research papers carrying out alkalisation relies on NaOH greatly due to its wide availability, good modification properties and economicity. Most fibres on treatment with NaOH greatly show better mechanical i.e tensile and impact strength. Varying the % of alkali concentration gives a variation of the mechanical properties.

Then we subjected the fibre to vacuum oven heating at 80 degree C for 2hrs. to remove moisture content

Thus we can see that dewaxing followed by alkalisation and vacuum oven drying, leads to beneficiary effects on the sisal fibre and with the furthermore preparation of composites we will see that the mechanical and dielectric properties of the fibre have improved so much. As for the characterisation testing, we started with FESEM followed by XRD and FTIR. After preparation of the composite we give it a 3 point bending flexural test which gives us the flexural and tensile strength of the composite.

After dewaxing has been done, the obtained sisal fibre was divided into 4 parts. One was kept as it is, the rest were treated to different concentration of alkalis, here it was KOH, which is supposed to enhance surface modification. The

treatments were respectively with 1% KOH(1 g KOH in 100 ml water), 3% KOH and 5% KOH solution respectively, all left therewith for 24 hours. After 1 day, fibres were washed and stored in different containers in a cool, dark place to protect it from humidity, moisture and other environmental cons.

Now our objective was to study the different analyses of the fibres which can give us a good overview of the different property enhancements of the fibres. We performed firstly (a) FESEM , followed by characterisation with (a) XRD (b) FTIR and after composite preparation wrapping up by (d) 3 pt. flexural test.

Lets see in brief what all these techniques are about and what they might predict

2. FESEM

FESEM(Field emission scanning electron microscope) is a microscope which relies on electrons instead of light.. Scanning is done by the electrons acc. to a zig-zag pattern used to see minute topographical details on the surface or total or fractioned objects. Electrons are ejected from a field emission source and accelerated in a high electric field gradient. The object is now bombarded by primary electrons in the high vacuum column, which have been focussed and deflected by electronic lenses. The angle and secondary velocity of these secondary electrons relates to the surface structure of the object. The secondary electrons helps in production of an electric signal with the help of a detector. This is then amplified and converted to an image, subjected to saving and further processing on a monitor.

Firstly, fine parts of the fibre less than 1 cm in length were made conductive by coating them with an extensively thin layer of platinum(1.5-3 nm). Cold slush of Nitrogen i.e cryo-fixation was also performed. The testing was done in the Ceramic department.

Magnification at different ratios were performed by utilising both ETD and TLD cameras with magnification ranging from 1000x-50 000x with test length varying from 4.3-4.5 mm.

We moved onto XRD next

3. XRD(X-Ray Diffraction) method

X Ray diffraction is an experimental technique which utilises Bragg's law to study the intensity vs 2 theta curve , thereby giving characteristic property of the sample.

About 0.2 g sample from each cluster was taken and powdered with the help of a ceramic pestle and mortar, until very finely grounded. Next the 4 batches were taken to the XRD Analysis machine in the Metallurgical Department, powered by Bruker© . Co was used as the source element.

The finely grounded samples were kept and on the other hand glass slides were cleaned using Acetone and cotton, followed by lubing it with Vaseline for better characterisation.

We can safely say from the analysis that

(a) XRD provided most definitive structural information regarding interatomic distances and bond angles.
(b) We need to probe atomic distances~probe wavelength in the order of Angstroms.
(c) Co is bombarded with a beam of electrons emitted from a tungsten filament. This ionises electrons from K-shell of the target atom and X-rays are emitted as resultant vacancies are filled by electrons dropping down from L or M levels. This gives rise to k_{α} and K_{β} lines.

Next we moved onto FTIR for further characterisation.

4. FTIR(Fourier transform infrared spectroscopy)

FTIR(Fourier Transform Infrared Spectroscopy) is an analytic technique used to identify polymers, organic or inorganic substances. Infrared light is used to scan samples and observe chemical properties. Basically FTIR identifies chemical bonds in a molecule by generating an IR spectrum. This method is effective in detecting functional groups and characterising covalent bonding information. FTIR studies the extensive H-bonding between cellulose chains, producing strong crystal structures. With aid of FTIR, researchers are able to obtain much more in-depth information of natural fibres after various modifications. It can also used to detect change in chemical composition of natural fibres due to inherent defect. Removal of hemicelluloses in dislocation regions may cause decrease of transfer of shear stress under tensile loading and loss of lignin as well.

Firstly we took the 4 batches of fibre in sizes less than 1 cm and took them to SPONCOM lab in the Mining department for making pellets. The pellets were prepared by mixing the grounded fibre with KBr and finely crushing them and subjecting them to high pressure resulting in formation of pellets. Then those were taken to Material characterisation lab in the Chemical Engineering department where the analysis was performed. Datas were generated using Excel sheet.

5. Composite formation

About 4 g from each batch of sample was taken and made into rectangular shaped hard-cases consisting of treated fibres matrixed with thermoset(here L-epoxy). L-epoxy resin with an amine type hardener was used here in the ratio 10:1 (here we took 100ml and 10 ml respectively). This resin is a class of reactive pre-polymers and polymers which contain epoxide groups. This may

cross-link with themselves through catalytic homo-polymerisation or with a wide range of co-reactants including poly-functional amines. This falls under the type of thermoset polymers. The L-epoxy used here has the following properties:-

(a) Low viscocity, free of solvent and fillers
(b) Fast impregnation of glass, aramid and carbon fibres
(c) High static and dynamic strength

L-epoxy is also the most used laminating and hardening resin.

Heat distortion temperature ranges from 60 degree to 95 degree centigrade.

The hardener here is a L-type amine hardener . Curing is achieved by reacting epoxy with itself or by forming co-polymers with hardeners. Amine type alters both the processing properties(viscocity and reactivity) and the final properties of the cured network.

After placing the composite's mixture within the desired framework, we leave it for 12 hours to get it hardened. Use of Si spray ensures hassle-free sticking and polythene has to be applied all around the composites mixture so that the corresponding composite comes in the form of a slab. The fibres were cut in sizes less than 1 cm for the preparation. After completion of the composite formation, we now channel it for 3 pt. bending test.

6. 3-Point Bending Flexural Test

The composites so formed were of the following dimensions:

(a) Raw--- 50x38x7.6 mm^3
(b) 1% KOH treated---50x30.2x6.7 mm^3
(c) 3% KOH treated---50x30.66x6.5 mm^3
(d) 5% KOH treated---50x37.47x6.1 mm^3

This test provides values for modulus of flexuration in bending E_f, flexural stress σ_f, flexural strain $€_f$ and flexural stress-strain relation of material.

Flexural stress is given by $\sigma_f = 3FL/2bd^2$

Strain is given by $€_f = 6Dd/L^2$, where symbols have their usual meanings.

Flexural modulus= stress/strain

The INSTRON Machine was as follows(fig 5):-

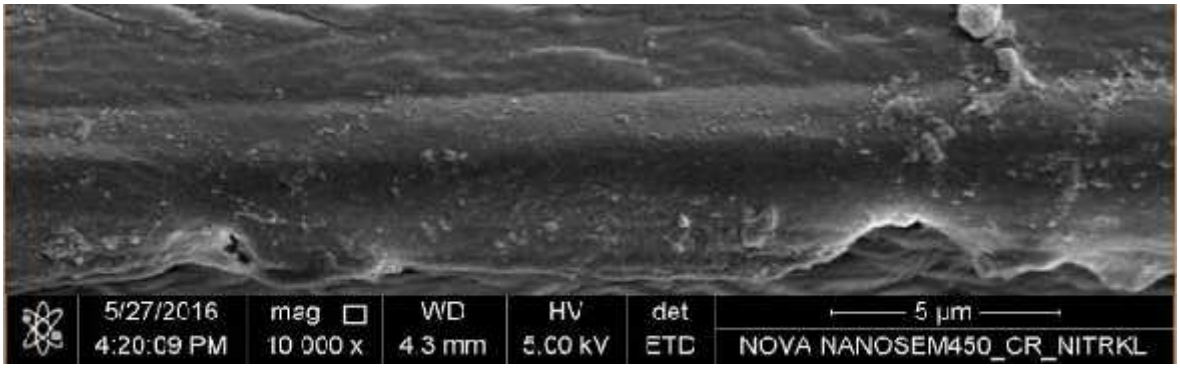
Now we move on to the results and discussions

CHAPTER 3

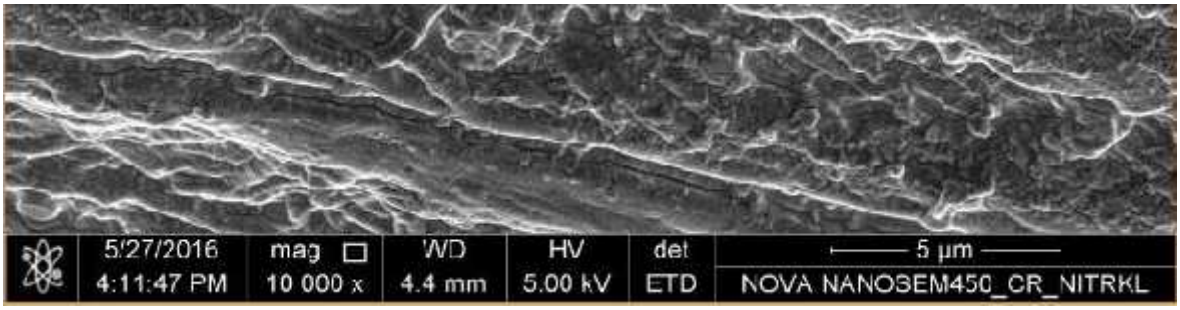
RESULTS AND DISCUSSIONS

(a)FOR FESEM

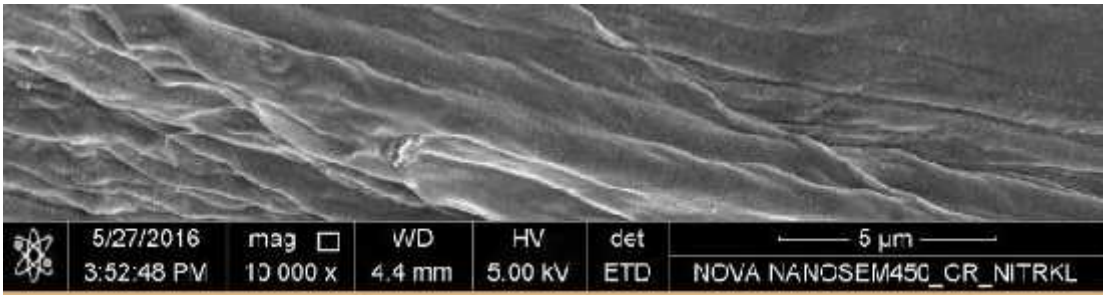
1. For Raw



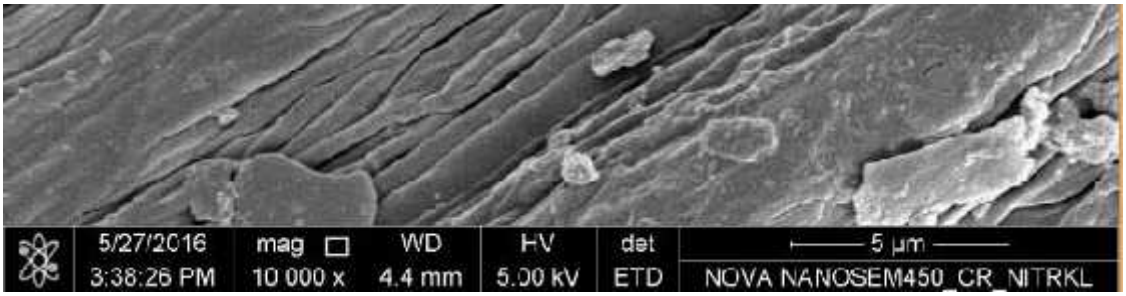
2. For 1% KOH treated sample



3. For 3% KOH treated sample



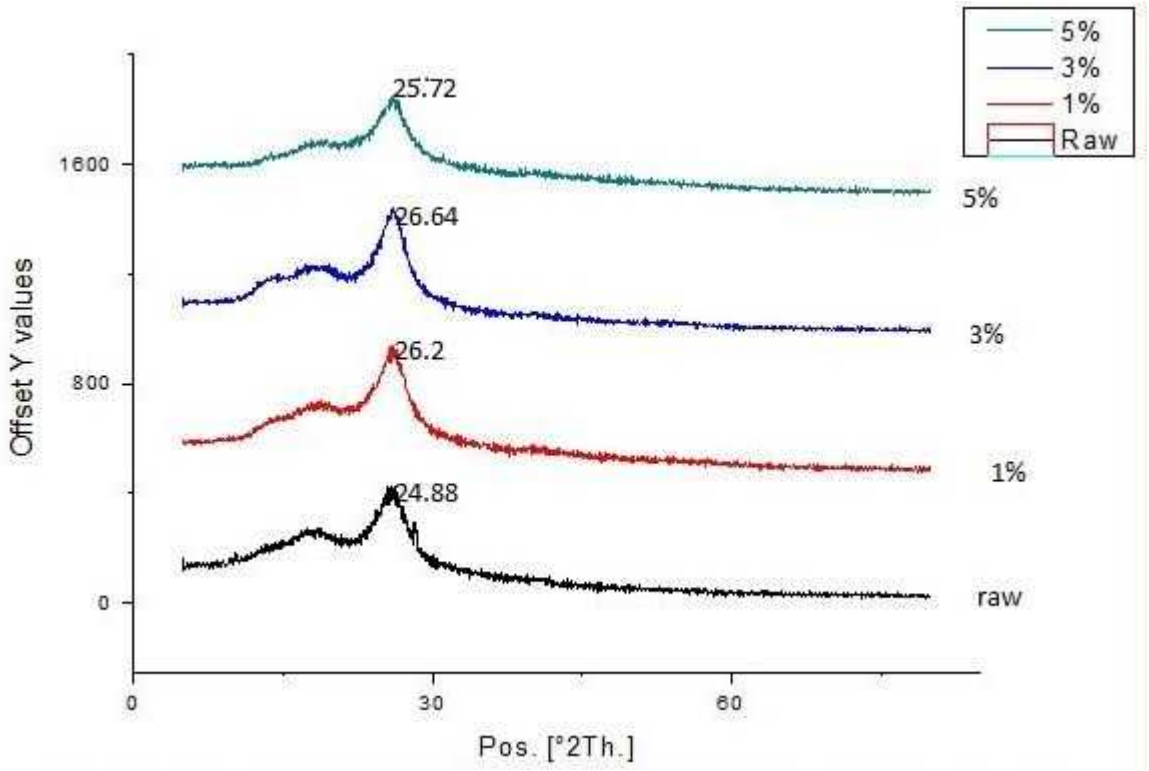
4. **For 5% KOH treated sample**



Discussion

So far we have seen, surface modification with the help of alkalisation leads to change in surface properties of the fibre. Raw and 1% samples aren't that much affected as 3% and 5% samples. 5 % treated ones are affected in big way, as rupturing starts as a result of excessive treatment with KOH . It has been watched that surface of crude sisal strands contrasts in smoothness and unpleasantness than synthetically treated sisal filaments. These micrographs plainly demonstrated the distinction in their surface morphology So in this regard 3% is the optimum one.

(b) FOR XRD



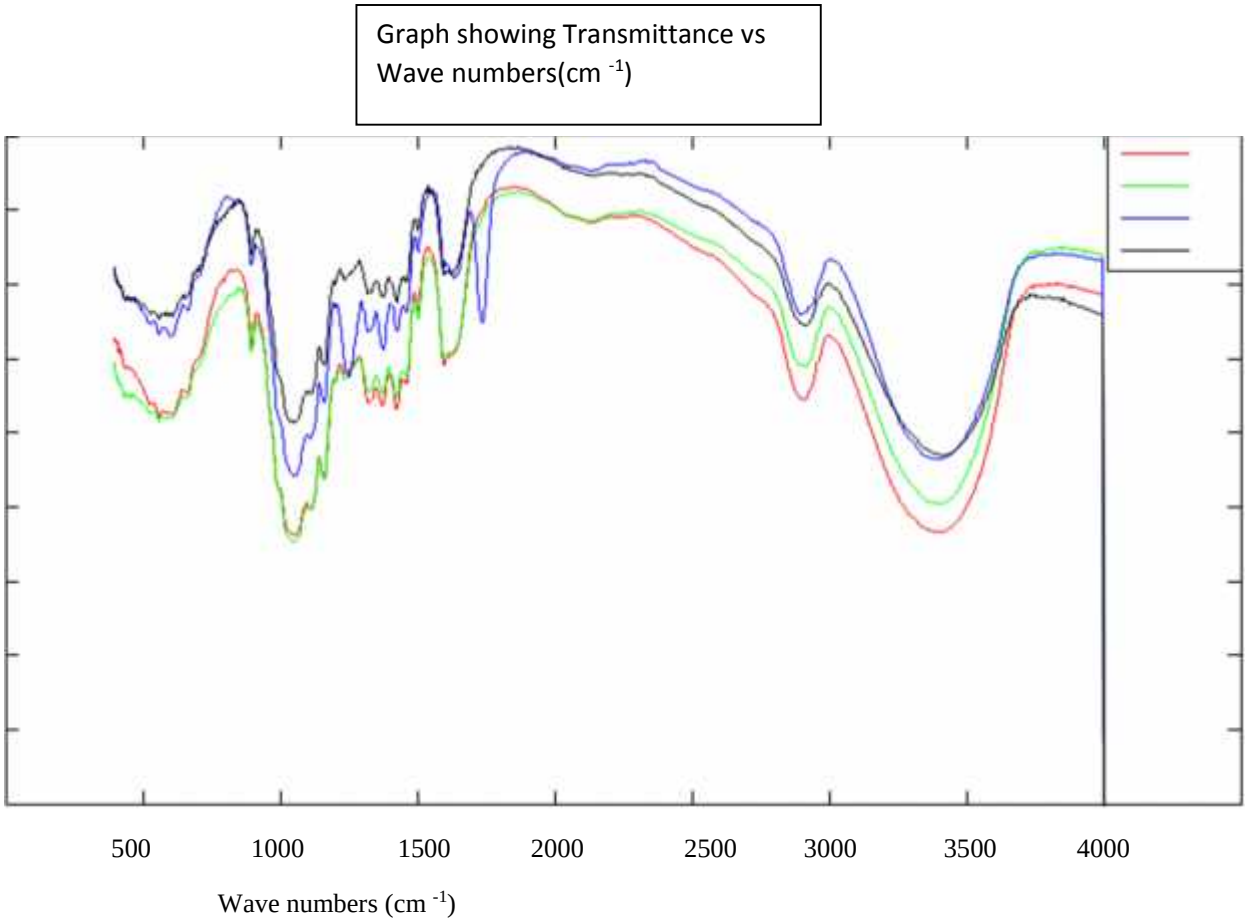
As suspected, from the intensity vs 2theta diffraction curve, we see that our fibre is mostly amorphous in nature. A poor crystallinity index, as shown by the curve, implies there should be an occurrence of alkali treated sisal strands implies poor output of cellulose crystals to the fiber pivot amid treatment, showed by the lower crystallinity file. Hence unmistakably show that cellulose precious stones are better situated in sisal filaments took after by alkali treated sisal strands . Peaks obtained at max. intensity for corresponding Bragg's angle develop a hump rather than sharp peak.

From Bragg's law,

$N\lambda = 2d \sin\Theta$

5% and 3% samples give max. intensity peaks at 26.08 degrees, hence these 2 samples are advised to be more useful in our work purposes. But crystallinity also decreases with subsequent additional treatment.

(c) **For FTIR**



Red line=1%, Green line=5%, Blue line=Raw, Black line=3%

Many bands present in the natural fibre vanishes after dewaxing

Given below is a table for peak wave nos. and bands attributed to it

Peak wave no.(cm^{-1})	**Most general value**	**Bands**
3300-3400	3327	OH stretching
2800-2900	2883	C-H symmetrical stretching
1700-1750	1724	C=O stretching vibration
1600-1650	1623	OH bending of absorbed water
1500-1550	1506	C=C aromatic symmetrical stretching
1400-1450	1423	HCH and OCH inplane bending vibration
1350-1400	1368,1363	In-the-plane CH bending

1300-1350	1325	S ring stretching
1250-1300	1259	G ring stretching
1150-1200	1152	C-O-C asymmetrical stretching
1000-1100	1046,1020	C-C, C-OH, C-H ring and side group vibrations
890-900	895	COC,CCO and CCH deformation and stretching
600-680	662	C-OH out of plane bending

Lets study the variations in wave no. corresponding to the different samples

Mean value	**Raw**	**1% KOH**	**3% KOH**	**5% KOH**
3327	-79.23	-79.1	-78.82	-78.9
2883	-20.29	-20.38	-34.81	-34.32
1724	-12.56	disappears	disappears	disappears
1623	-12.66	25.96	25.57	25.71
1506	0.52	2.2	3.43	2.64
1423	-5.61	-2.83	-3.43	-2.57
1368	-9.35	-5.21	-9.52	-7.51
1325	6.69	1.58	4.25	-3.01
1259	7.18	disappears	disappears	disappears
1152	-10.24	-9.99	-10.63	-8.72
1046	-10.22	-9.39	-10.14	-10.14
895	-2.15	-1.99	-0.73	-2.41
662	57.39	101.27	100.9	100.84

Conclusion

Also after alkalisation,for 1%, 3% and 5% samples, lack of band around 1250 cm^{-1} wave no. suggests absence of G band stretching and at 1274 cm^{-1} suggests absence of C=O stretching vibrations. The disappearance of the double bond stretching signifies more room for surface modification and better hydrophobic properties, becoming less averse to moisture and humidity. Inherent hydrophilicity of the fibres decrease quite a lot after treatment processes, as the surface gets rougher and more modified prohibiting linkage of –OH bonds.

(d) For 3 point bending flexural test

Test length for each sample was taken to be 50 mm

Under INSTRON conditions Machine Parameters were:

Sample rate (pts/s) = 4.552

Crosshead speed (mm/min) = 2

Full Scale Load Range (kN) = 50

Humidity (%)= 50

Temperature (deg F) = 73

1. Raw

Width(mm)	Depth(mm)	Load at peak(kN)	Displacement at peak(kN)	Stress at peak(MPa)	Strain at peak(mm/mm)
38	7.6	1.18	2.007	40.31	0.0366

From the formula modulus comes out to be= 40.31/0.0366= 1101.37

Flexural Stress=3 Fl/2 bd^2= 40.32 MPa

2. 1% KOH treated

Width(mm)	Depth(mm)	Load at peak(kN)	Displacement at peak(kN)	Stress at peak(MPa)	Strain at peak(mm/mm)
30.2	6.7	1.274	1.15	70.47	0.0185

Modulus=70.47/0.0185= 3809.19
Flexural Stress=3 Fl/2 bd^2=70.48 MPa

3. 3% KOH treated

Width(mm)	Depth(mm)	Load at peak(kN)	Displacement at peak(kN)	Stress at peak(MPa)	Strain at peak(mm/mm)
30.66	6.5	1.406	1.267	81.38	0.0198

Modulus= 81.38/0.0198 = 4110.1
Flexural Stress=3 Fl/2 bd^2=81.4 MPa

4. 5% KOH treated

Width(mm)	Depth(mm)	Load at peak(kN)	Displacement at peak(kN)	Stress at peak(MPa)	Strain at peak(mm/mm)
37.47	6.1	1.255	1.846	67.51	0.027

Modulus= 67.51/0/027= 2500.37
Flexural Stress=3 Fl/2 bd^2=67.5 MPa

Discussion

Amongst the 4 samples, the composite with 3% KOH treated condition gives the best values of flexural strength and flexural modulus, giving 81.4 MPa and 4110.1 respectively. Other samples,between 1 % and 5 % ones, the 1% sample unexpectedly gives better result than 5 %,which maybe due to the fact that rupturing in the 5 % sample leads to weakening of interfacial bonds resulting in decrease of flexural strength as a consequence.

FINAL CONCLUSION

Firstly we performed dewaxing, followed by alkalisation and vacuum oven drying. Then after performing FESEM, XRD , FTIR and a corresponding 3 pt. bending flexural test, we come to the conclusion that the 3% sample has been found out to be the most appropriate amongst the four . Morphology of sisal fibre was changed by chemical modifications. The surface of sisal fibre gets to be rougher after modifications in with smooth also, clear surface of crude sisal filaments. The evacuation of surface debasements on plant filaments might be leeway for fibre to grid attachment as it might encourage both mechanical interlocking and the holding response because of the introduction of the hydroxyl gatherings to chemicals, for example, dyes and colours. Almost in every analysis, 3% sample shows optimum results or near about. Theoretically 5% sample is predicted to be the best amongst the four but optimally keeping all in mind and working under the best provided scientific lab working conditions, 3% sample is most desirable.

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